BOND POTENTIAL OF LITHIUM DISILICATE TO HEAT-CURED POLYMETHYLMETHACRYLATE (PMMA)

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CERTIFICATE OF APPROVAL

MASTER'S THESIS

This is to certify that the Master's thesis of

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ABSTRACT

BOND POTENTIAL OF LITHIUM DISILICATE TO HEAT-CURED POLYMETHYLMETHACRYLATE (PMMA)
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Introduction: The hybrid prosthesis or implant-supported, implant-retained prosthesis utilizing acrylic prosthetic gingiva is an increasingly popular restorative design for complete or partially edentulous patients. The most common clinical mode of failure is a cohesive or adhesive failure between the denture tooth and the denture base. To improve success, lithium disilicate has been proposed as an alternative material for the prosthetic tooth. To date the restorative interface between lithium disilicate and polymethyl methacrylate (PMMA) is untested. **Objective**: To compare the bond potential between lithium disilicate and heat-polymerized PMMA with various surface treatments. **Method**: Lithium disilicate samples were divided into 4 groups (N=10) and received the following surface treatments; 1.) negative control (NC), no lithium disilicate surface treatment, 2.) hydrofluoric acid etch of lithium disilicate (HF), 3.) hydrofluoric acid etch and ceramic priming agent (HF+Primer) of lithium disilicate, and 4.) positive control (PC), milled polymerized PMMA with monomer wetting. The specimens were processed using a heat-polymerizing PMMA following manufacturer's instructions. Specimens were shear loaded on a static load testing machine (MTS Insight) at crosshead speed of 0.01mm/s. **Results**: The mean shear loads (MPa) were; PC, 13.65±1.5, NC, 7.1±2.8, HF, 21.1±3.0 and HF+ Primer 22.1±3.9. One-way ANOVA and a post hoc Bonferroni test

identified significant differences between groups, $p<0.001$, when comparing the positive and negative controls to each other, and when comparing either control to test group HF and test group HF+Primer. There was no significant difference between the test groups HF and HF+Primer. **Conclusions**: This study suggests that acid etching the ceramic is the most significant factor in achieving a bond between lithium disilicate and heat-polymerized PMMA. The addition of a ceramic priming agent to the surface of the ceramic may not aid in creating a stronger bond.

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Chapter 1: Introduction

The continued study of dental materials involved in the construction of denture prostheses remains important because denture prostheses and will always be in demand. Studies investigating denture use show although the percentage of dentures as a restorative option is declining, population growth is resulting in an increase in total denture wearers (National Center for Health Statistics, 1994). The number of US adults requiring complete dentures is anticipated to increase from 35.4 million in 2000 to 37.9 million in 2020 (Douglass et al 2002). Indeed properly constructed dentures have been shown to increase the quality of life for patients. Continued study on the strength and longevity of removable prostheses must be done to meet the needs of this group of patients (Critchlow et al 2010; Hummel et al 2002).

A common complication with removable prostheses is the de-bonding or fracture of teeth (Darbar et al 1994). The failure rate of arylic resin based prostheses due to fracture is suspected to be very high (Cunningham 1993, Vallittu et al 1993). Some studies have suggested as much as 30% of all denture repairs completed in dental laboratories involved failed bonding between denture teeth and the denture base resin (Huggett et al 1982, Vallittu et al 1993). Further complicating this common failure mode is the hypothesis that the use of implants in removable or fixed prosthesis designs will lead to even greater prevalence of resin failure. Due to the loss of normal mechanoreceptive input from teeth, implant-supported prostheses can produce increased

bite force and greater failures of denture teeth (Gunne et al 1997, Lindquist et al 1985). This suspicion was later supported by clinical findings showing the prevalence of tooth debonding from denture base resins or cohesive fracture through the denture tooth is one of the most common complications of implant-supported prostheses (Goodacre et al 2003, Walter et al 194, Davis et al 2003). It is clear from the literature that this restorative interface is highly susceptible to failure and improvement needs to be investigated to reduce the complication rates of acrylic-based restorative prostheses.

Chapter II: Review of the Literature

Denture Base Chemistry

PMMA is composed of a long carbon chain matrix which may contain other function groups to improve certain chemical or physical properties. The polymerization of PMMA undergoes the same process regardless of the type of polymerization: initiation, propagation and termination. Chemical-polymerizing PMMA and heat-polymerizing PMMA share similar initiators, namely, benzoyl peroxide. The activator, which decomposes the initiator to create free radials, is either a tertiary amine, which is used in chemical-polymerizing PMMA, or heat, which is used in heat-polymerizing PMMA. The exception to this is light activated resin polymers used in denture base resins such as urethane dimethacrylate (UDMA) with a photoinitiator system. Following initiation and the formation of free radicals of the monomers, propagation occurs during the creation of chains of monomers, forming a polymer, until the polymer is stericly hindered and termination of polymer formation occurs (Zarb et al 2004).

Additional chemical components alter the physical properties of the resin polymer. Plasticizers, namely dibutyl phthalate, increase monomer penetration into pre-polymerized PMMA beads during the mixing and early polymerization stages of the reaction. They have been shown to increase the stiffness and thermal diffusivity of the resin. Cross-linking agents such as glycol dimethacrylate increase the molecular weight of the polymer by covalently crosslinking long polymer chains, which aids in crack and crazing resistance of the resin. Many denture base resins are reinforced with butadiene styrene rubber, which serves to make the resin more impact resistant to fracture. High

impact resins were formulated to help prevent a common failure mode if the prosthesis is dropped on the ground by the patient during regular use (Rodford 1986). Other additives such as opacifiers and dies are used to change color and optical properties of the resin material (Zarb et al 2004).

Another common polymerization method is microwave-cured resins which have the same chemical constituents as heat-polymerizing resins. They have been shown to have comparable strength and durability and have the principle advantage of decreasing the processing time of polymerization (Al-Hanbali et al 1991, Sanders et al 1991, Polyzois et al 1987).

Light-polymerizing urethane dimethylmethacrylate (UDMA) has demonstrated similar mechanical properties to more traditional heat-polymerizing PMMA (Ali et al 2008, Machado et al 2007, Diaz-Arnold et al 2008). The resin system has the advantage that it eliminates flasking, boil-out and long processing times. All phases of the denture are composed of UDMA and cured using visible light in special curing ovens developed by the manufacturer.

Denture Teeth

Originally denture teeth were fabricated using high temperature fusing feldspathic porcelain and were used predominately until around the 1960s. Up until this time, porcelain was the only material available to meet the esthetic demands, but had some significant drawbacks. This type of porcelain had low facture toughness and was brittle. Due to the high abrasive nature of this formulation of ceramic, it was harmful to opposing

natural dentition (Hodson 1959, Bodicker 1947, Koran 1972). Patient's also commonly complained of clicking noises during function as they chewed to talked (Zarb et al 2004).

PMMA denture teeth were developed later and had some advantages over porcelain teeth. Chemical bonding was achievable between the PMMA denture tooth and denture base (Schoonover 1952, Spratley 1987, Suzuki et al 1990). They lacked the clicking noise attributed to porcelain teeth and were non-abrasive to natural tooth structure (Koran et al 1972, Harrison et al 1975, Eckfeldt et al 1989).

Denture Base Polymerization and Bonding

There are various denture base chemistries and laboratory techniques that may alter bonding potential of the denture tooth to the denture base. Microwave based polymerization techniques have shown conflicting results in terms of denture tooth bonding. The polymerization rate and excessive production of heat from the exothermic polymerization process can cause boil-out of the monomer, leading to porosity within the denture base (De Cleak 1987). Porosity can reduce the strength of resin materials and is a probable explanation of the difference in bond strength between denture bases and denture teeth seen in the literature during microwave processing techniques (Gettleman et al 1977, Polyzois et al 1993, Polyzios et al 1995, Keller et al 1985). Results from other studies have shown the opposite result, where bond strength between denture base and denture tooth was greater in microwave-polymerized resins compared to conventionally processed heat-polymerizing resins (Geerts et al 1993)

Compared to conventionally processed heat-polymerizing resins, visible light polymerizing resins have shown to decrease potential to bond to denture teeth (Clancy et

al 1991, Cunningham 2000). Investigators have hypothesized that the reason for this difference involves a reduced ability of the UDMA compatible monomer to wet the bonding surface of the denture tooth, however research in this area is still underway (Kawara et al 1991, Cunningham 2000).

Another possible variation is denture base to denture tooth bonding may involve laboratory procedures that do not optimally create an appropriate bonding surface (Huggett et al 1982). Stone-acrylic separating solution can prevent bonding if not cleaned from the surface of the denture tooth (Rupp et al 1971). Insufficient wetting of the denture tooth with monomer prior to processing or wax residue contamination on the denture tooth surface can also create weaker bonding potential (Cunningham et al 1999).

Interface Between Denture Base and PMMA Denture Teeth

Mechanical Retention

Research has continued to investigate means to increase retention of denture teeth. Micro and macro mechanical approaches have both been utilized. Cardash et al 1986 showed that diatorics or vertical retention indentations into the denture tooth, increase the retention of PMMA denture teeth to heat polymerized denture base. Chung et al 2008 investigated the effect of breaking the glaze layer on the denture tooth through grinding and air-particle abrasion using aluminum oxide particles and found a significant increase in bond strength compared to untreated controls. Moreover, they showed the highest bond strengths when the acrylic tooth surface was treated with grinding and air-particle abrasion combined. There is overwhelming evidence that surface modification in general increased the bond potential between PMMA denture teeth and PMMA denture bases.

Surface modifications included bur abrasion, diatorics, aluminum oxide particle abrasion and tribochemical coating (Cardash et al 1990, Nishigawa et al 2006, Consani et al 2010, Consani et al 2011, Vallittu et al 1997, Vallittu 2009).

Chemical Retention

A number of studies have investigated various chemical treatments to increase retention of PMMA teeth to PMMA and UDMA denture bases. Applying liquid methyl methacrylate monomer to the denture tooth and allowing it to wet the surface prior to flasking as demonstrated favorable increased retention in multiple studies (Geerts et al 1993, Barpal et al 1998, Rached et al 2001). The predominate theory behind these results is that the monomer creates a better wetting surface and/or partially dissolves the surface layer of the pre-polymerized denture tooth, allowing the denture base acrylic to diffuse and penetrate deeper into the tooth surface and create a stronger chemical bond (Papazoglou et al 2009, Barbosa et al 2009). Other chemical treatments including organic solvents such as dichloromethane, chloroform, acetone and methylene chloride have shown similar increased retention (Shen et al 1984, Hayakawa et al 1991, Rached et al 2001, Yanikoglu et al 2002, Rached et al 2004, Minami et al 2004).

Interface Between Denture Base and Silica-Based Ceramics

Silica-Based ceramics have been investigated as a replacement of PMMA denture teeth. The principle advantages of porcelain denture teeth were increased esthetic capability and reduced wear of the occlusal surface. However, due to the poor fracture toughness of predominately feldspathic ceramics, studies that investigated the bond

between porcelain and PMMA showed cohesive fractures within the ceramic (Paffenbarger et al 1967, Semmelman et al 1968). Myerson in 1969 theorized the polymerization shrinkage and resulting shear and tensile stresses applied during cooling of heat-polymerized PMMA lead to cohesive failures, despite observations that the actual bond between these earlier ceramics and PMMA was strong.

Silica-based ceramics can categorized in multiple ways, one of which is composition. They are feldspathic, leucite-reinforced, and lithium-disilicate ceramics (Kelly 2008). There silica-ceramics are etched with 5%-9.5% hydrofluoric acid for 20-60 seconds depending on the material and then treated with a silane coupling agent prior to bonding with a resin cement. It has been shown that etching the ceramic with acid increases the surface free energy and surface area available for bonding, which in turn increases the bonding ability of the ceramic surface (Chen et al 1998, Ahmad 2002). Bonding significantly increases the fracture strength of silica-based ceramics, which are otherwise very brittle and have poor fracture toughness (Blatz et al 2003, Kelly 2008).

Silane coupling agents are an important part of the bonding process. They are used to create covalent bonding between inorganic and organic substrates. One of the predominate silane coupling agents is 3-trimethoxysilylproprymethacrylate or (MPS). MPS is a bisfunctional molecule, which allows coupling between inorganic and organic substrates. The bonding mechanism is still theoretical however the bonding potential is believed to be increased with chemical bonding, increased wetting and through a condensation reaction (Clark et al 1963, Umemoto 1995, Matinlinna et al 2004). Silane molecules wet the surface of the inorganic ceramic and react with each other through hydrogen bonding between hydroxyl -OH groups. These siloxane bonds form through a

condensation reaction, -Si-O-Si-, which creates a siloxane polymer chain across the ceramic surface. Silica and metal oxides on the ceramic surface are also available through hydrogen bonding between hydroxyl -OH groups forming –Si-O-Si- and -Si-O-M- bonds (M equals metal). The organic functional group at the silane is a vinyl group which copolymerizes with the organic matrix or in this case, the resin cement (Matinlinna et al 2004). In a more recent study investigating bond potential between silica-based ceramics to PMMA, Kanie et al 2000, mixed MPS with PMMA monomer liquid at a ratio of 94/6 mol% PMMA/MPS respectively. They found the ceramic had good bond strength at greater than 20 MPa to PMMA when MPS as a silane coupling agent was used in the monomer liquid. The predominate failure mode was again cohesive through the relatively weaker ceramic.

Lithium disilicate is the newest of the silica-based ceramics and has the highest flexural strength (320-440 MPa) and fracture toughness (2.5-3 MPa) of any other material within that classification category (Albakry et al 2003, Deng 2003, Lawn et al 2004). Lithium disilicate may thus be strong enough to withstand the processing stresses inherent in processing denture base resins and may more accurately measure the potential bond strength between PMMA denture resin and silica-based ceramics.

Summary

Material limitations in acrylic resin based prostheses, such as dentures, continues to be a maintenance problem and failure mode. PMMA denture teeth have continual wear over time that can result in changes in the occlusal vertical dimension of the prosthesis and the balance of the occlusal scheme. Porcelain denture teeth have

considerable maintenance concerns in that they are brittle and can fracture easily. Both restorative designs offer advantages and disadvantages and both have specific maintenance concerns. Multiple techniques such as diatorics, grinding, monomer wetting, air-particle abrasion have all shown improvement in PMMA denture tooth retention but cannot affect the inevitable wear patterns associated with the material over time.

This study investigates lithium disilicate ceramics, which have significant improvements in material properties and demonstrate significantly higher fracture toughness. These favorable properties make lithium disilicate and possible material choice in classic denture tooth restorative options. To date, the bonding ability of lithium disilicate to heat polymerized PMMA has not been tested. The purpose of this study is to test the shear strength of the interface with various surface treatments against a positive control of simulated PMMA denture teeth to a heat polymerized acrylic resin.

Chapter III: Materials and Methods

This in vitro investigation continues the study and uses some of the data of Dr. James Linkous. An additional test group was added to the study to improve analysis of the data. Two experimental groups with different surface treatments were originally devised with 10 specimens in each group:

1. HF: Lithium disilicate etched with 4.8% HF acid.
2. HF + Primer: Lithium disilicate etched with 4.8% HF acid and application of MPS ceramic primer.

Two control groups were devised with 10 specimens in each group:

1. CC: Ceramic Control (negative control) with Lithium disilicate with no surface treatment
2. AC: Acrylic Control (positive control) with pre-polymerized PMMA, with ground surface treatment and monomer wetting

12.50mm diameter cylindrical lithium disilicate ingots (E.MAX, Ivoclar USA) were treated consistent to their test groups. A single lithium disilicate ingot was scanned with a white light scanner (Freedom Scanner, Intuitive milling USA) and pre-polymerized ingots of the same dimension were copy milled from a PMMA puck (PrimaTech, USA) using a 5-axis mill and acrylic milling burrs. Test group HF and HF + Primer received 4.8% HF acid etch for 20 sec followed by 60 seconds distilled water rinse. Both groups were placed in 90% isopropyl alcohol solution and in an ultrasonic for 5 minutes and dried. Test group HF + Primer then received an application of MPS with scrubbing for

30 seconds and allowed to air dry. The lithium disilicate control received no surface treatment. The positive PMMA control was milled from a pre-polymerized PMMA puck and the milling was considered to break the glaze layer of pre-polymerized PMMA and aid in bonding. The specimens were then wetted with liquid methyl methacrylate monomer and scrubbed for 60 seconds.

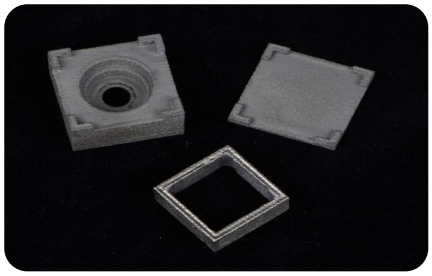


Figure 1: 3-D printed Titanium holder base, processing hold and cap

A 3-piece titanium metal processing apparatus was 3-D printed using the ARCAM A1 printer that uses electron beam melting technology (Fig. 1). The ceramic and PMMA were treated and placed into the base of the processing holder with a polyvinylsiloxane (PVS) spacer used to help aid in separation of the specimen from the holder base (Fig. 2). PMMA (Lucitone 199, Dentsply USA) was mixed according to the manufacturer's instructions and injected into the denture PMMA processing holder allowing the mixed PMMA to contact and surround the exposed flat surface of the lithium disilicate ingots and pre-polymerized PMMA specimens (Fig. 3).

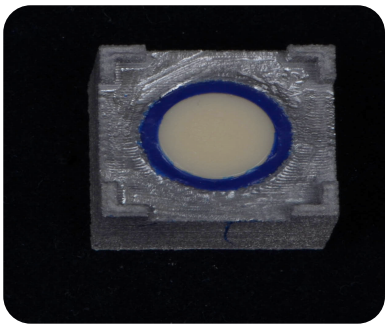


Figure 2: Treated sample placed into holder base and PVS injected as a spacer. Excess PVS was removed

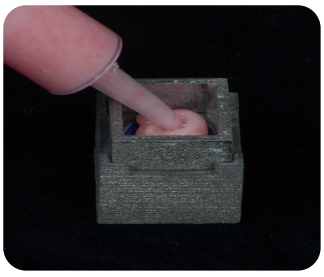


Figure 3: PMMA injection into the processing holder. Injected until overfilled and excess removed

A metal cover was placed on top of the metal processing apparatus and compressed to 30 psi and held together under pressure for 30 minutes (Fig.4). The processing apparatuses were placed

in a clamp and inserted into a water processing unit and heat polymerized under a slow cure protocol which consists of an 8 hour curing cycle at 165 degrees Fahrenheit with a final boil off temperature of 212 Fahrenheit for 1 hour.

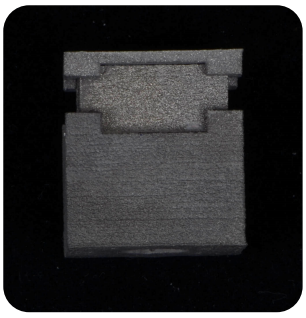
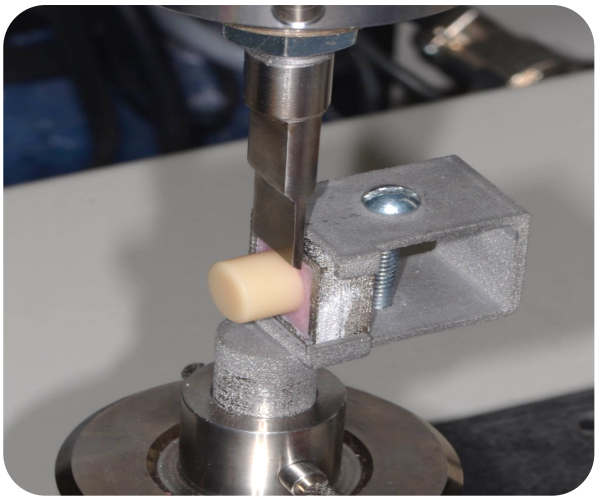


Figure 4: Assembled processing apparatus with excess PMMA removed



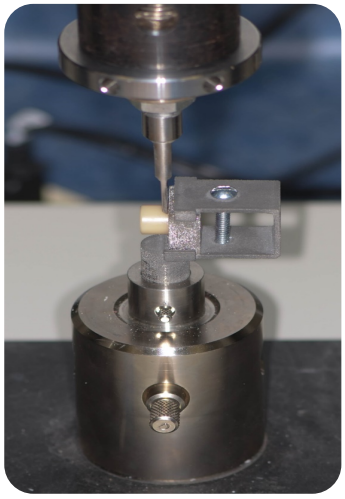


Figure 5 and 6: Test specimen bonded to processing holder positioned in the custom Titanium holder for the MTS Insight machine

Following processing, the specimens were removed from the processing apparatus and were hand assessed to be firmly attached to the square processing holder. The specimen attached to the processing holder was seated in the custom titanium holder and positioned within the MTS Insight machine so that the blade touched as close to the specimen-Denture PMMA interface (Fig. 5). The screw and nut was tightened to hand tight to secure the processing holder. Shear bond strength was tested and specimens were

loaded at the same location using a custom holder on an MTS Insight machine with a knife edge blade at a data acquisition rate of 60 hz, a preload of 1N, and a load speed of 0.01mm/s until breakage (Fig. 6).

Chapter IV: Results

Positive Control (PMMA-PMMA)	Negative Control	HF	HF+MPS
12.9	9.5	21.9	23.6
15.7	3.4	23.6	23.4
12.7	7.2	20.2	21.6
16.3	10.1	21.7	23
14.8	11.1	21.7	25.3
11.5	5.6	22.2	25.4
13.2	6.9	18.4	19.9
12.3	9.1	26.5	24.1
13.2	4.4	15.9	17.7
13.9	4	18.6	17.5

Table 1: Raw shear stress data in MPa

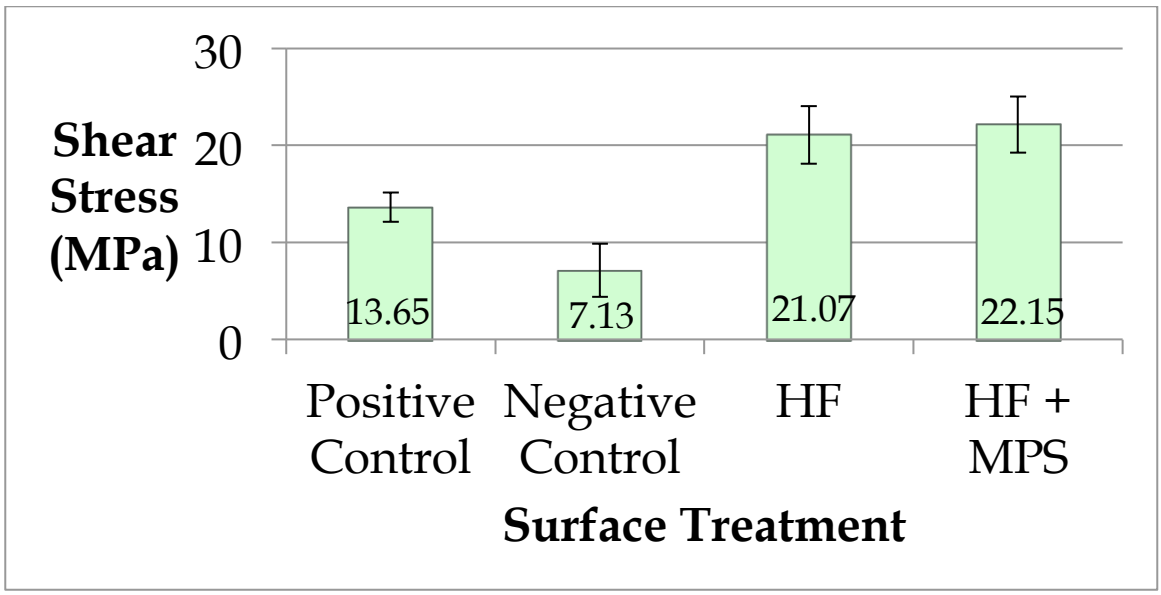


Figure 7: Mean shear stress: PC 13.65MPa +/- 1.5; NC 7.13MPa +/- 2.7; HF 21.07MPa +/- 2.9; HF+MPS 22.15MPa +/- 2.8

Raw data is listed in Table 1. The mean failure modes of the samples were different. The positive control group of PMMA to PMMA demonstrated a mixed adhesive and cohesive failure within all specimens. In all the ceramic samples, the failure mode was adhesive.

An Unpaired One-way Anova test was performed, resulting in a p-value less than 0.001. A Post hoc Bonferroni test was also performed, individually comparing the test groups. A p-value of less than 0.001 was obtained when comparing the positive and negative controls to each other, and when comparing either control to either the HF test group or the HF and Primer test group. There was no significant difference between the test groups HF and HF and Primer.

Chapter V: Discussion

The data showed evidence there is a bond potential between lithium disilicate and denture acrylic when the surface is enhanced and that the bond potential may be greater than the bond potential that exists between pre-polymerized PMMA and denture acrylic. Etching the ceramic was the most important factor in increasing bond potential based on this sample size. In all ceramic samples the shear test resulted in a facture at the ceramic-PMMA interface. This is the first this interface between a silica-based ceramic and PMMA has been adequately tested. Previous studies using weaker silica-based feldspathic ceramic resulted in cohesive fractures within the ceramic sample (Paffenbarger et al 1967; Semmelman et al 1968; Myerson RL 1969; Kanie et al 2000 and 2004).

Interestingly the data also showed no difference in bond potential of an etched ceramic when a ceramic primer like MPS was used. This was an unexpected result as typically silane coupling serves to increase ceramic bond strengths. It can be hypothesized that MPS may be inhibited by the acetone solvent in methylmethacrylate monomer, thus reducing the effectiveness of silane coupling. Another possibility is that adequate wetting of the ceramic surface was already achieved with HF acid etch and a functional silane layer offered no additional surface interaction. Such a result would suggest a predominately micromechanical bond instead of a partial chemical bond via the silane molecule. A third hypothesis may be that the application technique is significant. MPS was added to the surface of the test samples prior to acrylic processing. Previous work by Kanie et al (2000, 2004) have shown that the addition of certain silanes to the

denture methylmethacrylate monomer at a 6 mol% solution had a significant effect on the bond strength of PMMA to feldspathic porcelain compared to the untreated control. These findings suggest it may be necessary to add silane to the monomer to gain the chemical advantage of silane coupling during acrylic processing to ceramics. More research is needed to investigate that question.

Chapter VI: Conclusion

The purpose of this study was to examine the bond potential between lithium disilicate heat-polymerized polymethylmethacrylate. Acid etching following manufacture's recommendations for lithium disilicate had the most significant effect on increasing the shear bond potential to acrylic. MPS silane was not shown to have a synergistic effect, although more research is needed to investigate why. This data is in concordance with previous studies involving feldspathic porcelain and for the first time the ceramic-acrylic interface was challenged without the ceramic sample fracturing. Although more research is needed, lithium disilicate may be a viable material choice for hybrid prostheses where adequate macro and micromechanical retention design is achievable.

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